Quality control of non-nutritive sweeteners with NIR spectroscopy

NIRS gives mixture analysis results within one minute

The use of non-nutritive sweeteners as sugar substitutes in foodstuffs has risen dramatically in the last decade, e.g., in soft drinks and snacks. Two examples are sucralose, a halogenated sucrose derivative, and Stevia, derived from the leaves of the *Stevia rebaudiana* plant. Both are much sweeter than sugar and are used in much lower concentrations in foodstuffs. To ensure food safety, regulations for non-nutritive sweeteners are becoming stricter. Several analytical methods are available to determine various sweeteners using, e.g., high-performance liquid chromatography (HPLC), ion chromatography, and thin-layer chromatography. However, these methods are time-consuming and incur high running costs. Near-infrared spectroscopy (NIRS) allows the simultaneous determination of several sweeteners in less than one minute without any chemicals or sample preparation.

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Mixtures of both Stevia (0.5–4.5%) and sucralose (0.5–4.5%) in sucrose (95%) were prepared and analyzed to create a prediction model for quantification.

Samples were measured with a Metrohm NIRS DS2500 Solid Analyzer (**Figure 1**) using 15 mm disposable vials, a DS2500 holder, and a DS2500 Iris in reflection mode. The Metrohm software package Vision Air Complete was used for all data acquisition and prediction model development.

Table 1. Hardware and software equipment overview.

Equipment	Article number
DS2500 Solid Analyzer	2.922.0010
DS2500 Iris	6.7425.100
Disposable vials, 15 mm	6.7402.110
Vision Air 2.0 Complete	6.6072.208

Figure 1. Metrohm NIRS DS2500 Solid Analyzer used to determine Stevia and sucralose content in sucrose mixtures.

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All measured Vis-NIR spectra (**Figure 2**) were used to create a prediction model for quantification of sucralose and Stevia in sucrose. The quality of the prediction models was evaluated using correlation diagrams which display a very high correlation between the Vis-NIR prediction and the reference values. The respective figures of merit (FOM) display the expected precision of a prediction during routine analysis (**Figures 3–4**).
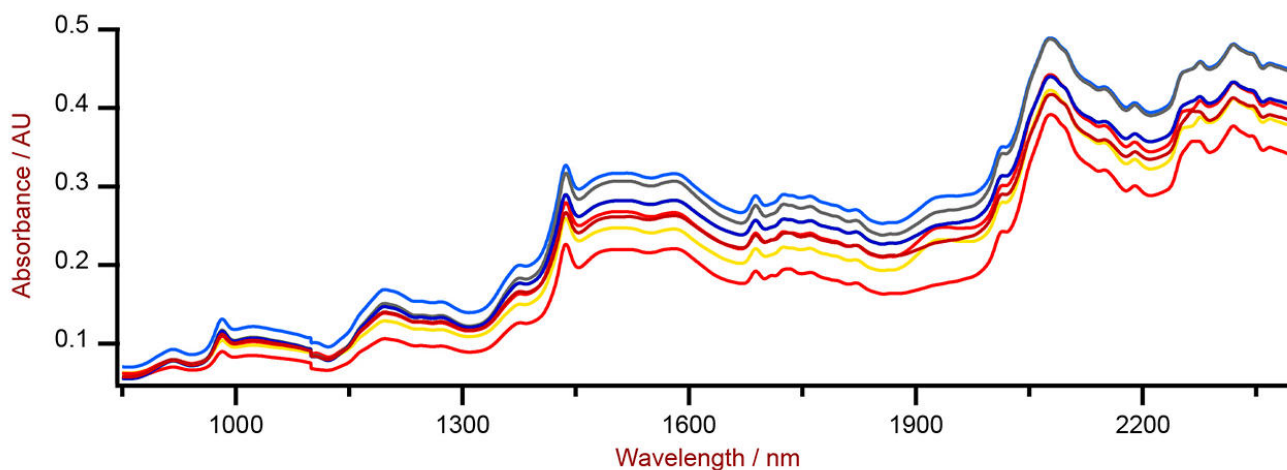

Figure 2. Selection of Vis-NIR spectra of Stevia and sucralose in sucrose samples which were analyzed on a DS2500 Solid Analyzer.

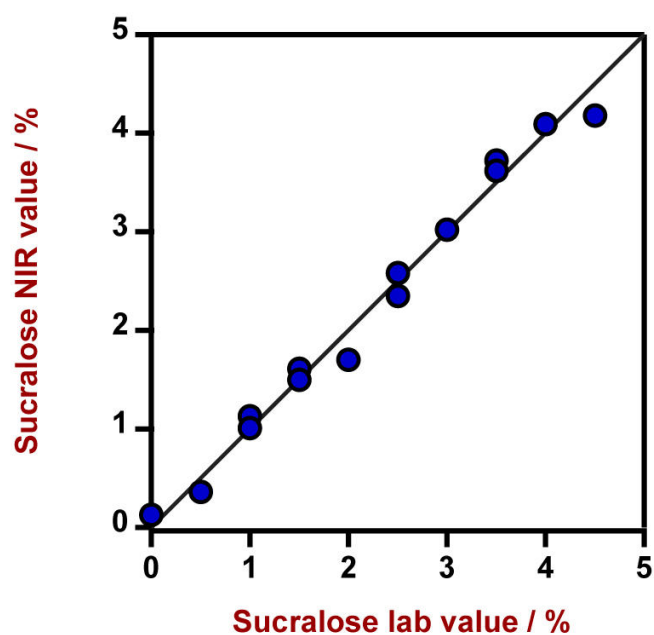Ω **Metrohm**

RESULT SUCRALOSE CONTENT IN SUCROSE

Figure 3. Correlation diagram and the respective figures of merit for the prediction of sucralose content in sucrose using a DS2500 Solid Analyzer. The lab values were determined using HPLC.

Figures of Merit	Value
R2	0.9854
Standard Error of Calibration	0.1898%
Standard Error of Cross-Validation	0.1997%

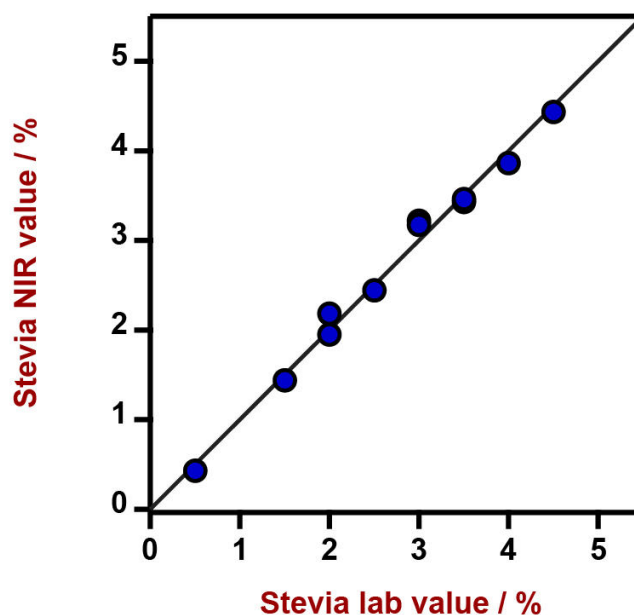Ω Metrohm

RESULT STEVIA CONTENT IN SUCROSE

Figure 4. Correlation diagram and the respective figures of merit for the prediction of Stevia content in sucrose using a DS2500 Solid Analyzer. The lab values were determined using HPLC.

Figures of Merit	Value
R2	0.9885
Standard Error of Calibration	0.1500%
Standard Error of Cross-Validation	0.1997%

CONCLUSION

This Application Note demonstrates the feasibility to determine the concentration of the non-nutritive sweeteners sucralose and Stevia in sucrose blends with NIR spectroscopy. Vis-NIR spectroscopy enables fast and cost-effective measurements with high accuracy, thereby offering a suitable alternative to other standard analytical methods (Table 2).

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Table 2. Time to result overview for the different non-nutritive sweeteners examined in this study.

Parameter	Method	Time to result
Stevia	HPLC	5 min (preparation) + 40 min (HPLC)
Sucralose	HPLC	5 min (preparation) + 40 min (HPLC)

Internal reference: AW NIR AE10-0002-072021

CONTACT

Metrohm USA
9250 Camden Field Pkwy
33578 Riverview, FL   info@metrohmusa.com

Table 2. Time to result overview for the different non-nutritive sweeteners examined in this study.

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CONFIGURATION

DS2500 Solid Analyzer

Robust near-infrared spectroscopy for quality control, not only in laboratories but also in production environments.

The DS2500 Analyzer is the tried and tested, flexible solution for routine analysis of solids, creams, and optionally also liquids along the entire production chain. Its robust design makes the DS2500 Analyzer resistant to dust, moisture, vibrations, and temperature fluctuations, which means that it is eminently suited for use in harsh production environments.

The DS2500 covers the full spectral range from 400 to 2500 nm and delivers accurate, reproducible results in less than one minute. The DS2500 Analyzer meets the demands of the pharmaceutical industry and supports users in their day-to-day routine tasks thanks to its simple operation.

Thanks to accessories tailored perfectly to the instrument, optimum results are achieved with every sample type, no matter how challenging it is, e.g. coarse-grained solids such as granulates or semi-solid samples such as creams. The MultiSample Cup can help improve productivity when measuring solids, as it enables automated measurements of series containing up to 9 samples.

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